OIPE JC98 JAN 12 2005 PATENT & TRADEMARK OFFICE

DOCKET NO: 217522US0PCT

IN THE UNITED STATES PATENT & TRADEMARK OFFICE

IN RE APPLICATION OF	:
TADAHIKO FURUTA, ET AL.	: EXAMINER: SHEEHAN, J.
SERIAL NO: 10/019,283	: GROUP ART UNIT: 1742
FILED: JANUARY 2, 2002	: RCE FILED: JUNE 24, 2004
FOR: TITANIUM ALLOY MEMBER	:

DECLARATION UNDER 37 C.F.R. § 1.132

COMMISSIONER FOR PATENTS
ALEXANDRIA, VIRGINIA 22313-1450

SIR:

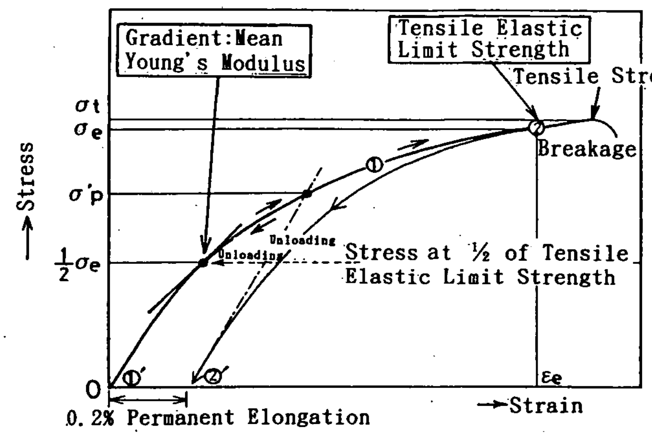
I, Tadahiko FURUTA, a citizen of Japan, hereby declare and state that:

1. I graduated from Suzuka College of Technology in 1982.

2. I have been employed since 1982 by Kabushiki Kaisha Toyota Chuo Kenkyusho, where I have been engaged in Materials Science and Engineering.

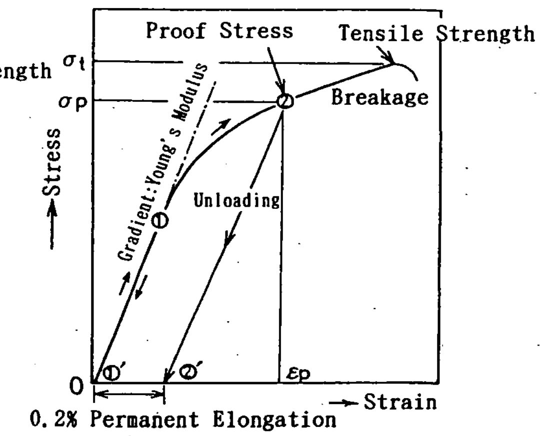
3. FIGS. 14A-14B of the above-identified application, which are reproduced below, compare the stress-strain curve of a conventional titanium alloy with the stress-strain curve of the titanium alloy of the present invention.

FIG. 14A



Titanium Alloy of Present Invention

FIG. 14B



Conventional Titanium Alloy

Conventional titanium alloys exhibit a linear stress-strain relationship at low stresses. In contrast, the titanium alloy of the present invention exhibits a non-linear stress-strain relationship at low stress. In particular, the titanium alloy member of the present invention has a tensile deformation property such that a gradient of the tangential line in a stress-strain diagram obtained by a tensile test within an elastic deformation range, in which the stress ranges from 0 to the tensile elastic limit strength, decreases continuously with increase in stress. The non-linear characteristic is achieved in the titanium alloy of the present invention by controlling the composition of the alloy and by cold-working the alloy.

4. Independent Claim 1 of the above-identified application is rejected as obvious, with U.S. Patent No. 5, 871,595 ("Ahmed") serving as the primary reference. Ahmed at, e.g., column 4, lines 56-60, discloses a conventional titanium alloy produced by working a cast ingot obtained by melting together pure elements. However, Ahmed is silent about cold-working the alloy and about the stress-strain characteristics of the alloy.

5. To compare Ahmed's conventional Ti alloy with the Ti alloy of the present invention having non-linear stress-strain characteristics at low stress, the following experiments were carried out by me or under my direct supervision and control.

Experimental Method

6. Sample corresponding to TA22 of Ahmed:

A sample corresponding to TA22 of Ahmed was produced by a melting method (or a sample was an ingot material). Specifically, Ti sponge, Zr sponge, small blocks of Ta and small blocks of Nb were used as raw materials. These raw materials were combined in proportions corresponding to a Ti-35.3Nb-4.9Ta-7.2Zr alloy, which corresponds to Ahmed's TA22. After compression molding, a briquette was prepared. The briquette was melted in an arc melting furnace under an argon atmosphere. The melt was cooled and cured in a water-cooled, copper mold to form an ingot.

The ingot was subjected to hot forging at a temperature of 1050°C, and a wrought product that was ϕ 10 mm x 100 mm was prepared. After carrying out a solution treatment at a temperature of 900°C for 30 minutes, a test piece having a plane portion that was ϕ 2 mm x 10 mm was prepared. The test piece was subjected to a tensile test at room temperature. The tensile test was carried out using an Instron universal testing machine at an early strain rate of 5 x 10^{-4}/sec. Young's modulus was estimated by attaching a strain gage to each test piece.

Neither this sample, nor the sample associated with Ahmed 's TA22 alloy in the Declaration Under 37 C.F.R. § 1.132 filed November 28, 2003, was cold-worked.

7. Sintered Ti alloy:

Samples were produced by a power metallurgy method using raw powders of Ti, manufactured by TOHO TITANIUM (TC459 (-#350)), and of Nb, Ta, and Zr, manufactured by KOUJUNDOKAGAKU (-#350). The raw powders were first mixed to form a mixed

powder. Each mixed powder was fully agitated by a shaker and then subjected to rotational mixing in a rotational ball mill mixing vessel for 2 hours. After that the mixed powder was filled in a silicon rubber mold and compacted by a CIP pressing machine at a pressure of 4 ton/cm^2. Then sintering was carried out in a vacuum of 10^{-5} torr at 1300°C for 16 hours. After the sintering, hot forging was carried out at 1050°C. After carrying out a solution heat treatment at 900°C for 30 minutes, cold working with 90% reduction in area was carried out, and then a test piece having a plane portion of 2mm × 10mm was prepared. Each test piece was subjected to a tensile test at room temperature. The tensile test was carried out by using an Instron universal testing machine at an early strain rate of 5×10^{-4}/sec. Young's modulus was estimated by attaching a strain gage to each test piece.

8. Cast Ti alloy ingot:

An ingot method (EB: electron beam melting) was used as a method for preparing a raw material. Specifically, as a raw Ti powder, TC459 (-#350) manufactured by TOHO TITANIUM and Nb, Zr (-#350) manufactured by KOUJUNDOKAGAKU were used. Ti powder, Nb powder and Zr powder were scaled to be Ti-30Nb-10Ta-5Zr-0.4O (mass%), and after that, the mixed powder was prepared. The mixed powder was subjected to rotational mixing in a rotational ball mill mixed vessel for 2 hours. After that, the mixed powder was filled in a vessel, and molded by a CIP pressing machine at a pressure of 4 ton /cm^2. Sintering was carried out in a vacuum at 10^{-5} torr and 1300°C for 4 hours. After sintering, hot forged processing was carried out at 1050°C. A cogging sample that was ϕ 10mm x 100mm was prepared. As preparation for melting, the pressure in a chamber was first reduced to about 10^{-3} Pa, and this degree of vacuum was maintained during melting. The cogging sample was supplied to the chamber from a transverse direction. An electron beam from a first electron beam gun (output: 70-80 kW) was applied to melt the cogging sample, and melting was carried out continuously. Simultaneously, molten metal in a hearth was

heated and poured into a mold. The molten metal in the mold was heated by a second electron beam gun (output: 30-40 kW). Then the temperature of the molten metal was continuously decreased, so that the molten metal was cooled and cured in the mold (made of copper), which was cooled by water, thereby obtaining an ingot. The speed of melting was 20kg/h.

A wrought product that was ϕ 13mm was prepared from the ingot, and a solution heat treatment was carried out at 900°C for 30 minutes. After that, the wrought product was subjected to cold working by a cold swaging machine and swaged to ϕ 4mm (cold working ratio 90%). A test piece having a plane portion that was ϕ 2mm x 10mm was prepared from the cold working sample, and the test piece was subjected to a tensile test at room temperature. The tensile test was carried out by using an Instron universal testing machine at an early strain rate of 5×10^{-4}/sec. Young's modulus was calculated by attaching a strain gage to each test piece.

As a comparative sample (see FIG. F), a test piece having a plane portion that was ϕ 2mm x 10mm was prepared from a non-cold working sample (a sample of ϕ 13mm was subjected to a solution heat treatment, but not subjected to cold working), and the same tensile test as above was carried out.

Furthermore, not only the electron beam method but also VAR (Vacuum Arc Remelting) was used as the melting method. In VAR, the same property as that of the electron beam method was obtained.

Experimental Results

9. FIG. D compares the stress-strain curve of the cast and hot forged Ti alloy ingot corresponding to the TA22 sample of U.S. Patent No. 5,871,595 ("Ahmed")(composition, in weight percent, Ti-35.3Nb-4.9Ta-7.2Zr) with the stress-strain curves of two sintered and

cold-worked Ti alloys of the present invention (compositions, in weight percent, Ti-38Nb-2Ta-2Zr-0.3O and Ti-30Nb-10Ta-5Zr-0.3O). The compositions of the two sintered and cold-worked Ti alloys of the present invention straddle the composition of Ahmed's TA22 alloy. FIG. D shows that Ahmed's TA22 cast and hot forged alloy exhibits a linear stress-strain curve at low stress. In contrast, the sintered, cold-worked Ti alloys of the present invention exhibit a non-linear stress-strain curve at low stress.

10. FIG. E shows the stress-strain curves for two cast Ti alloy ingots (compositions, in weight percent, Ti-30Nb-10Ta-5Zr-0.4O and Ti-36Nb-2Ta-3Zr-0.3O) after cold working. The two cold-worked Ti ingots exhibit the non-linear stress-strain curve at low stress characterizing the Ti alloy of the present invention.

11. FIG. F compares the stress-strain curve of a cast Ti alloy ingot (composition, in weight percent, Ti-30Nb-10Ta-5Zr-0.3O) before and after cold working. The non-cold-worked sample exhibits a linear stress-strain curve at low stress. In contrast, the cold-worked sample exhibits a non-linear stress-strain curve at low stress. FIG. F indicates that cold working is important for obtaining the non-linear stress-strain curve at low stress characterizing the Ti alloy of the present invention.

12. A comparison of the stress-strain curves in FIGS. D, E and F shows that cold-working produces a non-linear stress-strain curve at low stress in both cast Ti alloy ingots and sintered Ti alloys corresponding to the Ti alloy composition of the present invention. The comparison also shows that without the cold-working, the non-linear stress-strain curve at low stress of the present invention is not achieved. Ahmed's TA22 cast and hot forged alloy does not exhibit the non-linear stress-strain curve at low stress of the present invention.

13. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of this application or any patent issuing thereon.

14. Further declarant saith not.

Date: ______________________________ ____

Tadahiko FURUTA

Attached: FIGS. D, E and F

FIG. D

Comparison between Cited Reference and Ti Alloy of Present Invention

Present Invention: Sintered Method + Cold Working

Cited Reference: Melting according to Cited Reference

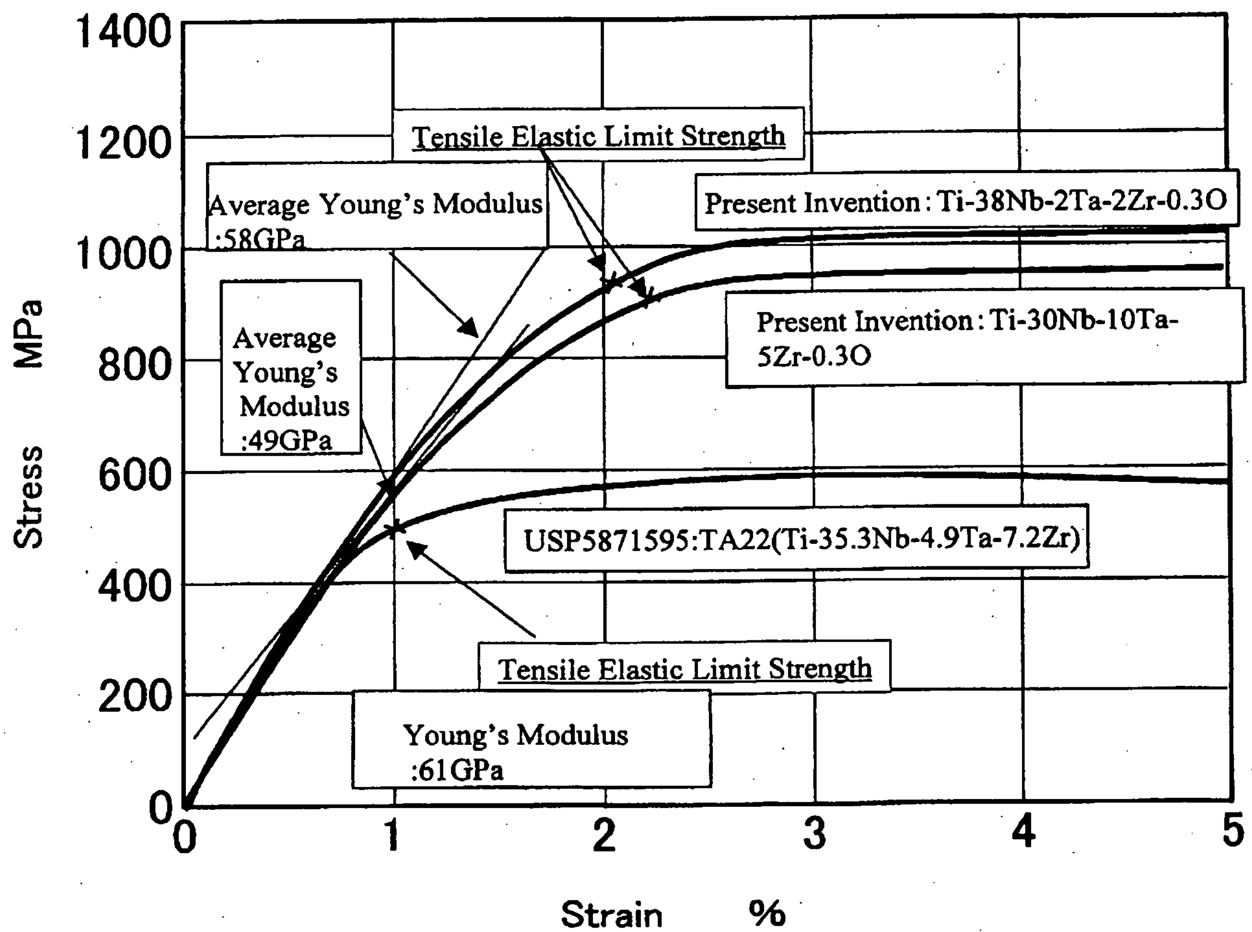


FIG. E

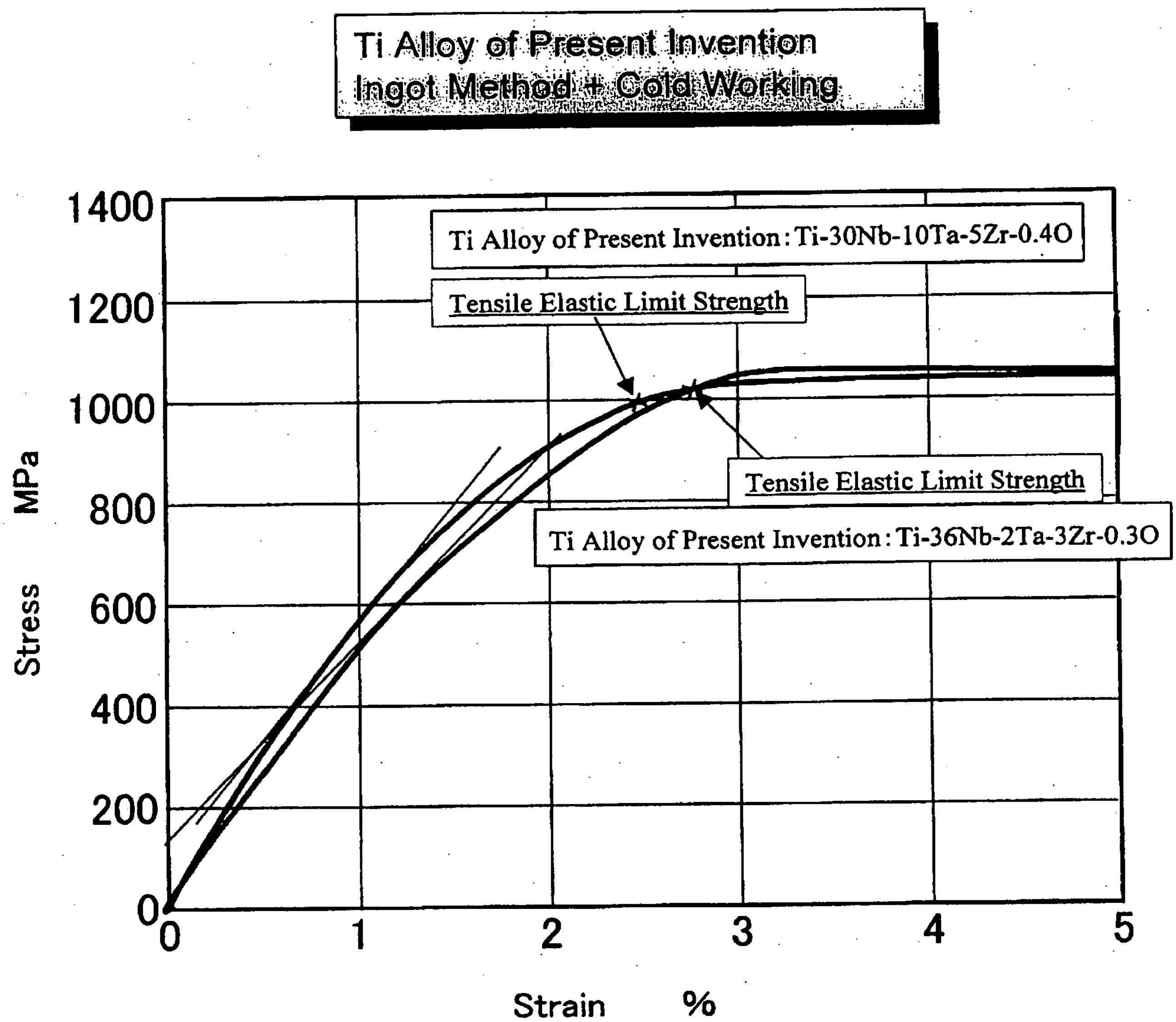

Ti Alloy of Present Invention
Ingot Method + Cold Working
Ti Alloy of Present Invention: Ti-30Nb-10Ta-5Zr-0.4O
Tensile Elastic Limit Strength
Tensile Elastic Limit Strength
Ti Alloy of Present Invention: Ti-36Nb-2Ta-3Zr-0.3O
Stress MPa
1400
1200
1000
800
600
400
200
0
0
1
2
3
4
5
Strain %

FIG. F

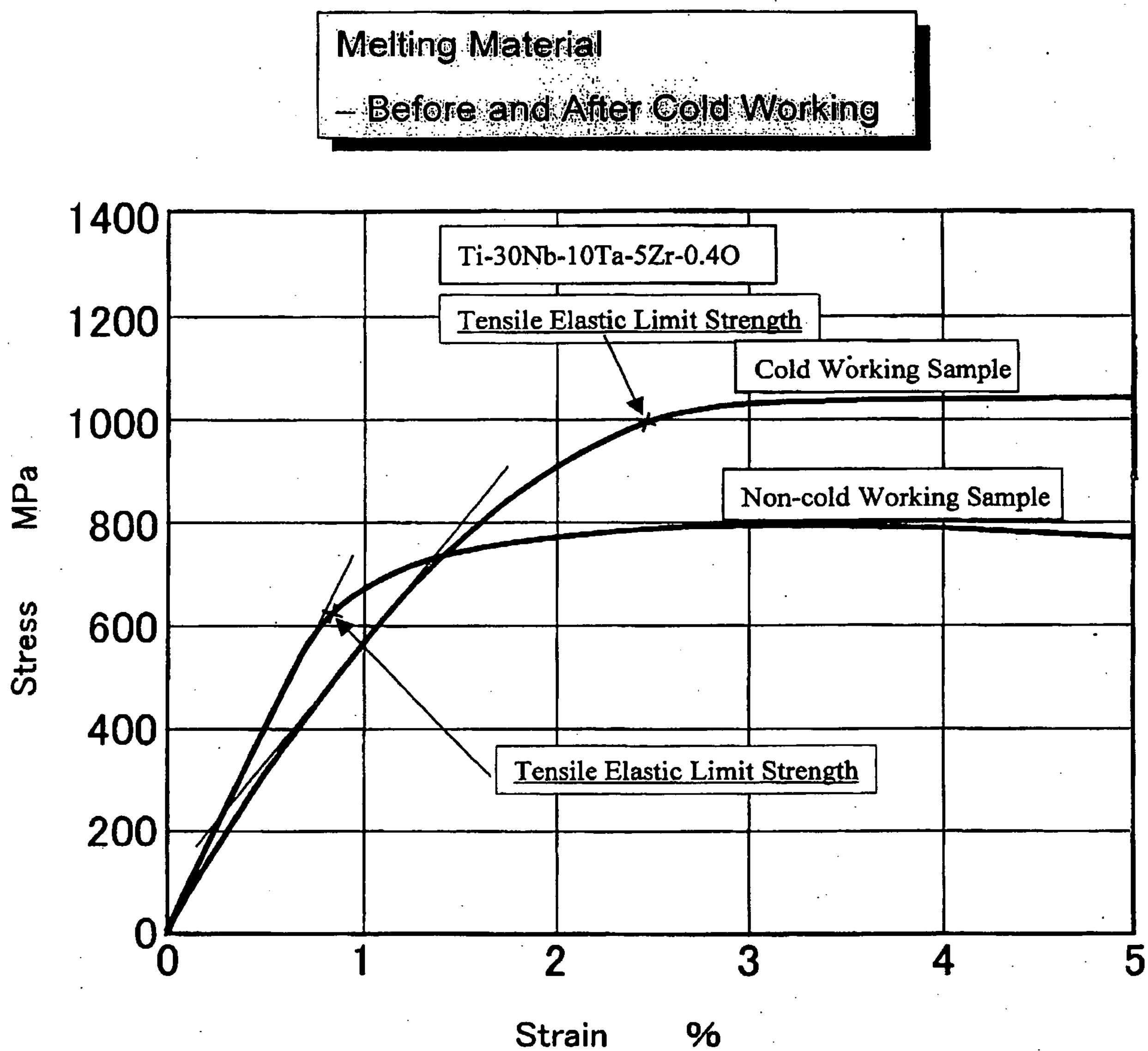

Melting Material
– Before and After Cold Working
Ti-30Nb-10Ta-5Zr-0.4O
Tensile Elastic Limit Strength
Cold Working Sample
Non-cold Working Sample
Tensile Elastic Limit Strength
Stress MPa
Strain %
0
200
400
600
800
1000
1200
1400
0
1
2
3
4
5

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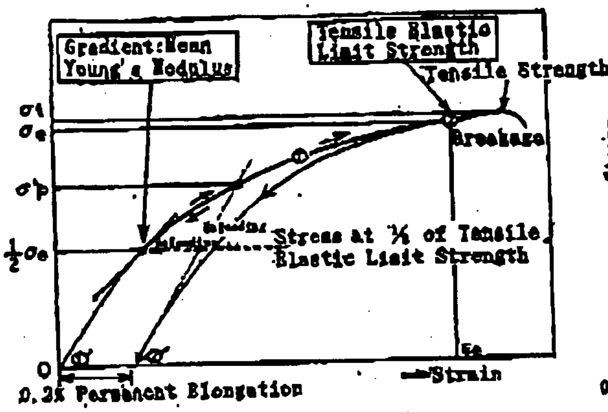
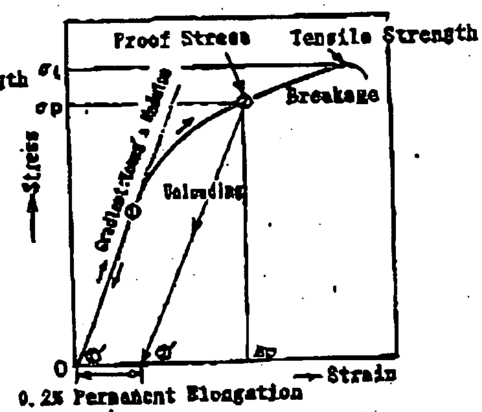
FIG. 14A



Titanium Alloy of Present Invention

FIG. 14B



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The ingot was subjected to hot forging at a temperature of 1050°C, and a wrought product that was ϕ 10 mm x 100 mm was prepared. After carrying out a solution treatment at a temperature of 900°C for 30 minutes, a test piece having a plane portion that was ϕ 2 mm x 10 mm was prepared. The test piece was subjected to a tensile test at room temperature. The tensile test was carried out using an Instron universal testing machine at an early strain rate of 5×10^{-4}/sec. Young's modulus was estimated by attaching a strain gage to each test piece.

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heated and poured into a mold. The molten metal in the mold was heated by a second electron beam gun (output: 30-40 kW). Then the temperature of the molten metal was continuously decreased, so that the molten metal was cooled and cured in the mold (made of copper), which was cooled by water, thereby obtaining an ingot. The speed of melting was 20kg/h.

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As a comparative sample (see FIG. F), a test piece having a plane portion that was ϕ 2mm x 10mm was prepared from a non-cold working sample (a sample of ϕ 13mm was subjected to a solution heat treatment, but not subjected to cold working), and the same tensile test as above was carried out.

Furthermore, not only the electron beam method but also VAR (Vacuum Arc Remelting) was used as the melting method. In VAR, the same property as that of the electron beam method was obtained.

Experimental Results

9. FIG. D compares the stress-strain curve of the cast and hot forged Ti alloy ingot corresponding to the TA22 sample of U.S. Patent No. 5,871,595 ("Ahmed")(composition, in weight percent, Ti-35.3Nb-4.9Ta-7.2Zr) with the stress-strain curves of two sintered and

cold-worked Ti alloys of the present invention (compositions, in weight percent, Ti-38Nb-2Ta-2Zr-0.3O and Ti-30Nb-10Ta-5Zr-0.3O). The compositions of the two sintered and cold-worked Ti alloys of the present invention straddle the composition of Ahmed's TA22 alloy. FIG. D shows that Ahmed's TA22 cast and hot forged alloy exhibits a linear stress-strain curve at low stress. In contrast, the sintered, cold-worked Ti alloys of the present invention exhibit a non-linear stress-strain curve at low stress.

10. FIG. E shows the stress-strain curves for two cast Ti alloy ingots (compositions, in weight percent, Ti-30Nb-10Ta-5Zr-0.4O and Ti-36Nb-2Ta-3Zr-0.3O) after cold working. The two cold-worked Ti ingots exhibit the non-linear stress-strain curve at low stress characterizing the Ti alloy of the present invention.

11. FIG. F compares the stress-strain curve of a cast Ti alloy ingot (composition, in weight percent, Ti-30Nb-10Ta-5Zr-0.3O) before and after cold working. The non-cold-worked sample exhibits a linear stress-strain curve at low stress. In contrast, the cold-worked sample exhibits a non-linear stress-strain curve at low stress. FIG. F indicates that cold working is important for obtaining the non-linear stress-strain curve at low stress characterizing the Ti alloy of the present invention.

12. A comparison of the stress-strain curves in FIGS. D, E and F shows that cold-working produces a non-linear stress-strain curve at low stress in both cast Ti alloy ingots and sintered Ti alloys corresponding to the Ti alloy composition of the present invention. The comparison also shows that without the cold-working, the non-linear stress-strain curve at low stress of the present invention is not achieved. Ahmed's TA22 cast and hot forged alloy does not exhibit the non-linear stress-strain curve at low stress of the present invention.

13. I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of this application or any patent issuing thereon.

14. Further declarant saith not.

Date: Jan. 6, 2005

Tadahiko FURUTA

Tadahiko FURUTA

Attached: FIGS. D, E and F

FIG. D

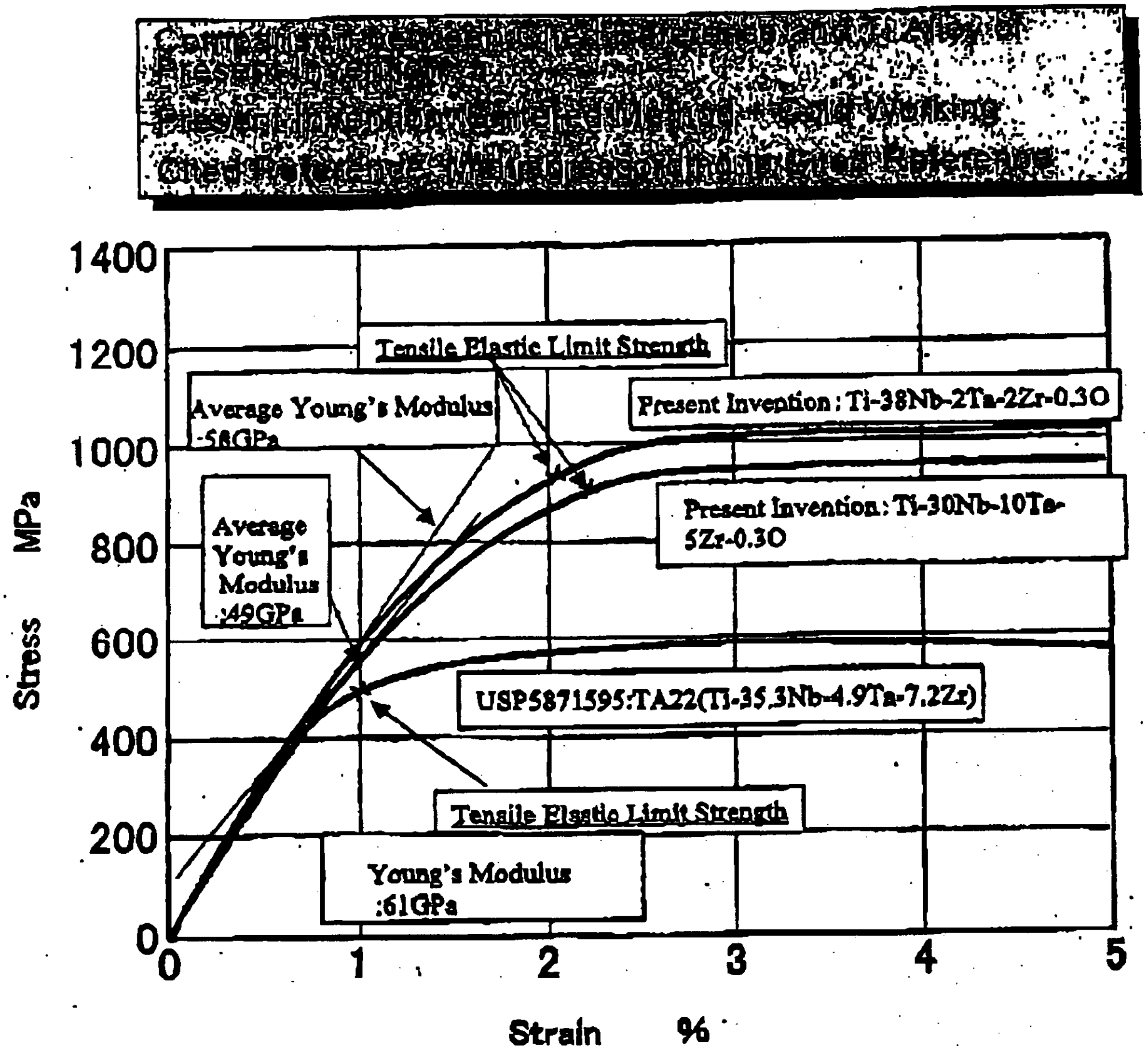

Tensile Elastic Limit Strength
Average Young's Modulus :58GPa
Present Invention : Ti-38Nb-2Ta-2Zr-0.3O
Average Young's Modulus :49GPa
Present Invention : Ti-30Nb-10Ta-5Zr-0.3O
USP5871595:TA22(Ti-35.3Nb-4.9Ta-7.2Zr)
Tensile Elastic Limit Strength
Young's Modulus :61GPa
1400
1200
1000
800
600
400
200
0
Stress MPa
0
1
2
3
4
5
Strain %

FIG. E

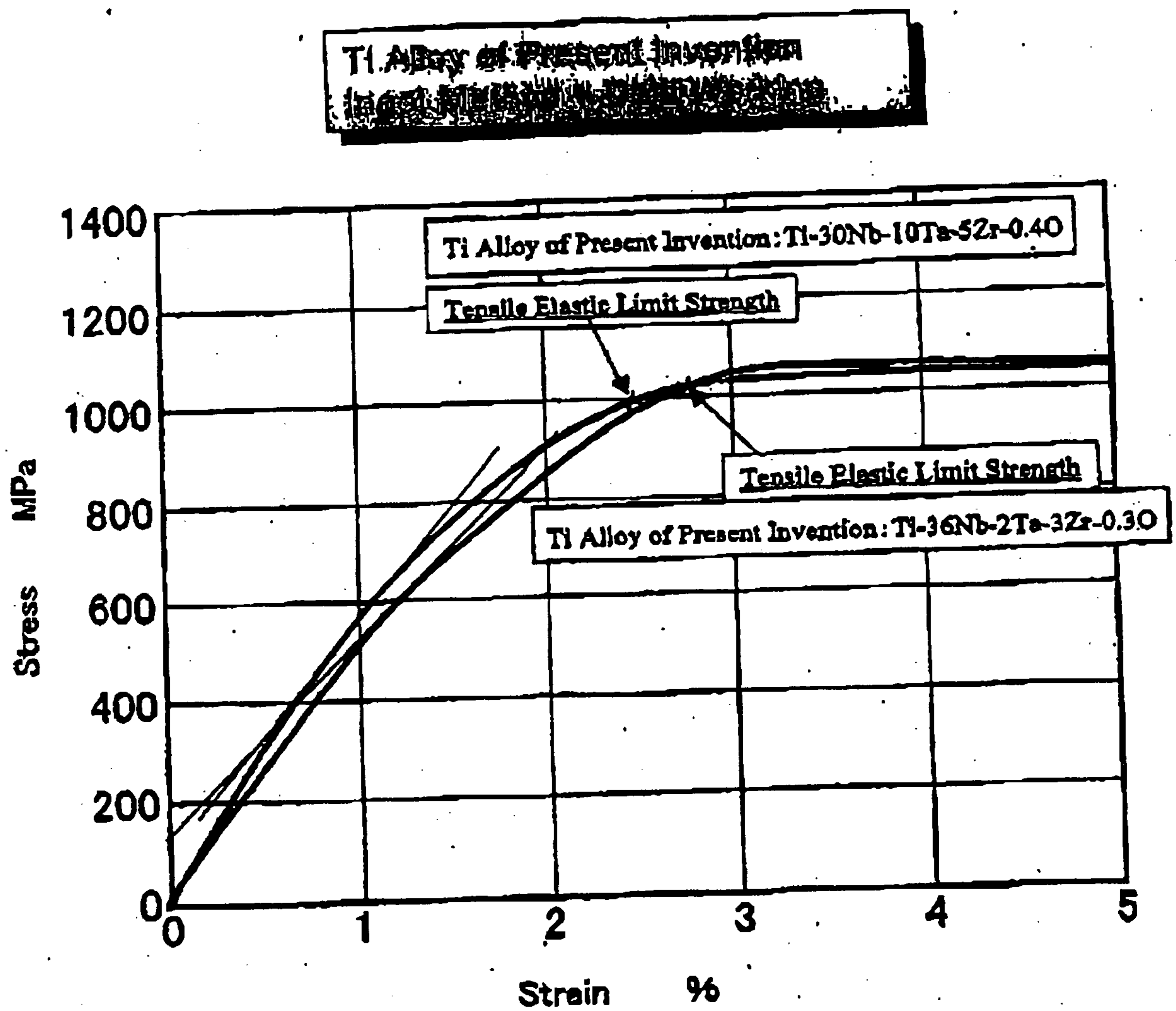
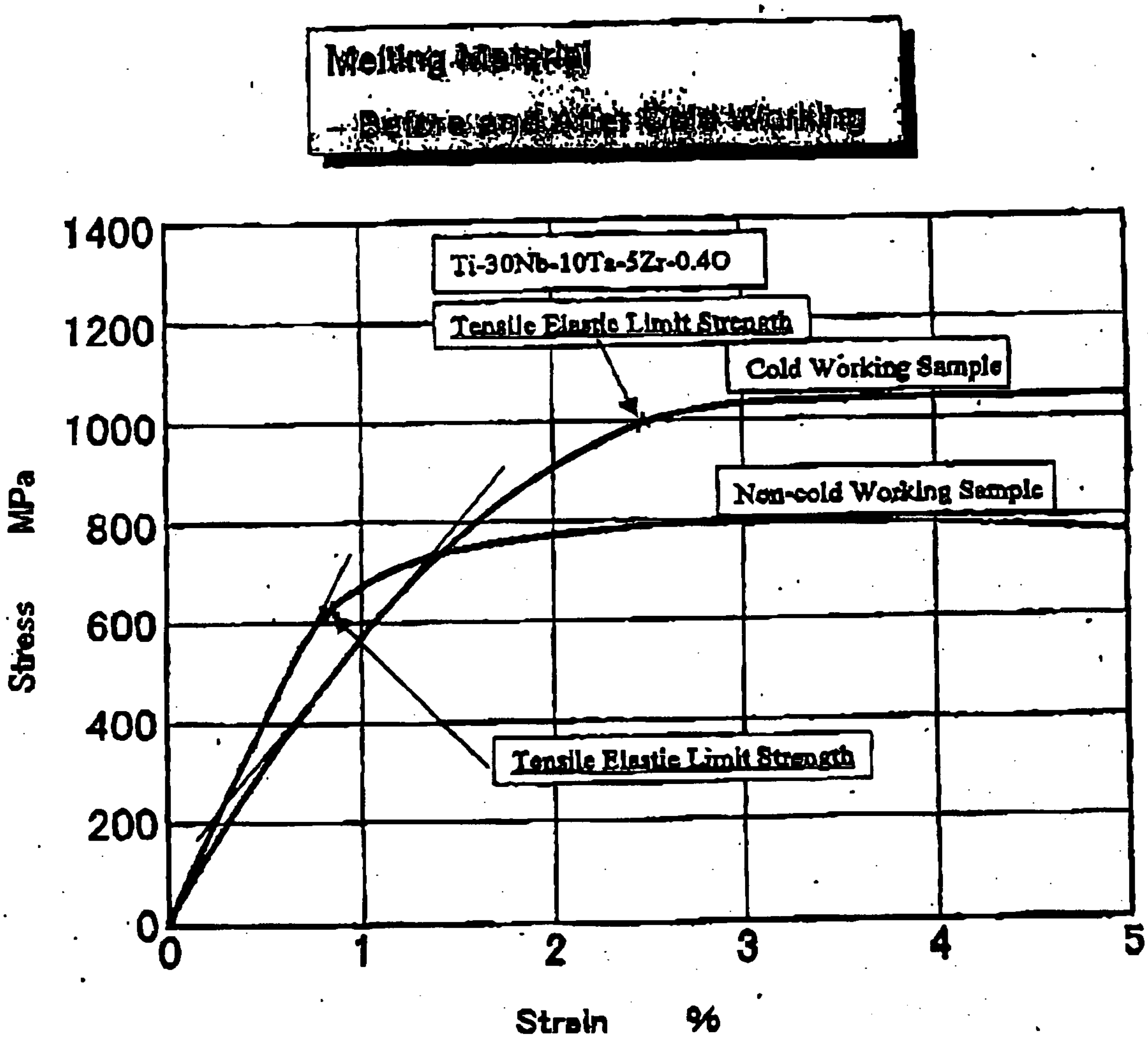

Ti Alloy of Present Invention
Ti Alloy of Present Invention: Ti-30Nb-10Ta-5Zr-0.4O
Tensile Elastic Limit Strength
Tensile Elastic Limit Strength
Ti Alloy of Present Invention: Ti-36Nb-2Ta-3Zr-0.3O
1400
1200
1000
800
600
400
200
0
Stress MPa
0
1
2
3
4
5
Strain %

FIG. F


Ti-30Nb-10Ta-5Zr-0.4O
Tensile Elastic Limit Strength
Cold Working Sample
Non-cold Working Sample
Tensile Elastic Limit Strength
1400
1200
1000
800
600
400
200
0
Stress MPa
0
1
2
3
4
5
Strain %